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### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

14

Applicant's or agent's file reference WO 800148-AI		See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) <b>FOR FURTHER ACTION</b>	
International application No. PCT/NL00/00525	International filing date (day/month/year) 24/07/2000	Priority date (day/month/year) 23/07/1999	
International Patent Classification (IPC) or national classification and IPC A23G1/18			
Applicant STICHTING VOOR DE TECHNISCHE WETENSCHAPPEN et al.			

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.


2. This REPORT consists of a total of 4 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  16/02/2001	Date of completion of this report  19.10.2001
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  MARZANO MONTERO..., M  Telephone No. +49 89 2399 2902



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/NL00/00525

## I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):
- Description, pages:**

1,4-9 as originally filed

2,3,3a as received on 04/10/2001 with letter of 03/10/2001

### Claims, No.:

1-6 as received on 04/10/2001 with letter of 03/10/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

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5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes:	Claims	1-6
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-6
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-6
	No:	Claims	

2. Citations and explanations  
**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/NL00/00525

**Item V:**

1. None of the documents in the available prior art discloses a method for the manufacture of chocolate comprising all the features of claim 1.
  - 1.1 Document WO 98 30108 A (D1) discloses a method according to the preamble of claim 1 wherein the seed material consists in powders of stable crystals of a fat or oil. Such seeding agents are different than what is disclosed in the present application wherein seed material is actually cooled mixture at a temperature above 30°C.
  - 1.2 Document DE 2602877 A (D2) discloses a method for the manufacture of chocolate wherein the seeding material is in fact cooled mixture. On the other hand, it is specified that the temperature is not higher than 30°C, so that in fact what is mixed with the chocolate mass is a mixture where crystallization kernels are already present.
  - 1.3 It must be concluded that even a combination of the features of the two documents would not lead to, nor hint at, a method according to claim 1. Said claim is therefore considered to meet the requirements of Art. 33(2) and (3) PCT with regards to novelty and inventive step.
2. Claims 2-6 are dependent on claim 1 and as such also meet the requirements of Art. 33(2) and (3) PCT with regards to novelty and inventive step.

liquid chocolate mass, it is heated to above the critical temperature, and subsequently cooled to a second temperature between the first temperature and the critical temperature, the thus cooled chocolate mass is mixed with the seed material, the seed material used being a cooled mixture at a temperature above 30°C, but which substance has not exceeded the critical temperature and which substantially does not contain any crystalline material in the  $\beta'$  phase, and in that to produce solid chocolate, the mixture is subsequently cooled to the first temperature.

Thus a simplified method is provided for the preparation of chocolate which during prolonged storage will exhibit less fat bloom or none at all. Compared with the known method, the energy consumption is limited. In the present application the critical temperature is the temperature at which all forms of crystalline fat have changed to the molten state. This temperature may be determined by melting a sample whose fat is in the  $\beta$  condition (obtained, for example, by leaving molten chocolate for at least three days at a temperature of 22°C) and to heat it to a temperature X and to maintain it for one minute at that temperature. The mass is subsequently cooled to 23°C at a rate of 1°C/min. (while avoiding that the liquid chocolate mass comes into contact with a surface whose temperature is more than 3°C lower than that of the chocolate mass), and examined to see whether the  $\beta$  or the  $\beta'$  phase develops. This experiment is carried out mechano-statically. That temperature X is the critical temperature with which after cooling again solid chocolate is obtained whose crystallization phase is substantially  $\beta'$ . The melting point of chocolate depends to some extent on the rate of cooling during production. Moreover, the melting point is not one single value, because chocolate has a melting range of several degrees. Indications of temperature with regard to melting temperature are in the present application related to the lowest value of the melting range. For a reliable process, the liquid chocolate mass will generally be heated to at

least 2°C, preferably to at least 5°C above the critical temperature. A higher temperature shortens the time during which the chocolate mass has to be heated above the critical temperature. In the present application, the first temperature is understood to be the temperature at which molten chocolate mass is solidified. This temperature is below the melting temperature of the chocolate. The second temperature, which may also be called the mixing temperature, is suitably at least 2°C below the critical temperature and at least 2°C above the melting temperature of the chocolate. Cocoa butter is preferably mixed at a temperature in the vicinity of  $T_{opt}$  obtained with the (empirical) formula:

$$T_{opt} = 1.44 * [St] - 3.3 * [Ar] - 6$$

[St] being the concentration of stearic acid and [Ar] the concentration of arachidic acid as present in the free ester form in cocoa butter. When the present application refers to a substance not having exceeded the critical temperature, this means counting from the last time the substance is at least partially in a crystalline ( $\beta$ ) phase.

As an alternative to ultrasonic treatment it is known in the art to add seed crystals to a liquid chocolate mass (Hachiya, I., et al. in Seeding Effects on Solidification Behaviour of Cocoa Butter and Dark Chocolate. II. Physical Properties of Dark Chocolate, in *J. Amer. Oil Chem. Soc.* 66:1763-1770 (1989)).

Such a method has several disadvantages. First, solid chocolate has to be ground into (preferably the smallest possible) seed crystals. Second, said seed crystals should be mixed as homogeneously as possible with the liquid chocolate mass. Until now, these disadvantages have prevented the application of this method in large-scale production of chocolate, which term includes in the present invention also chocolate-comprising products such as biscuits with chocolate, and the like. Preferably, in the cooling steps, the temperature of the wall (that is to

CLAIMS

1. A method for the manufacture of chocolate, which method comprises

a) the preparation of a cooled but still liquid chocolate mass which comprises i) a fat selected from  
5 cocoa butter and cocoa butter equivalents (CBE), and at least one component selected from a) sugar, b) cocoa mass and c) cocoa powder,

b) mixing the liquid chocolate mass with a seed material, and

10 c) allowing the mixture to cool to a first temperature below the melting temperature of chocolate, producing solid chocolate,

the seed material used in step b) being cooled mixture, characterized in that when preparing the liquid  
15 chocolate mass, it is heated to above the critical temperature, and subsequently cooled to a second temperature between the first temperature and the critical temperature, the thus cooled chocolate mass is mixed with the seed material, the seed material used being a cooled  
20 mixture at a temperature above 30°C, but which substance has not exceeded the critical temperature and which substantially does not contain any crystalline material in the  $\beta'$  phase, and in that to produce solid chocolate, the mixture is subsequently cooled to the first temperature.

25 2. A method according to claim 1, characterized in that the quantity of liquid substance being added is 10 - 20% by volume of the fat content of the final mixture.

3. A method according to claim 1 or 2,  
30 characterized in that prior to being mixed with the seed material, the liquid chocolate mass is cooled to a second temperature of at least 4°C below the critical temperature.

4. A method according to one of the preceding  
35 claims, characterized in that cooling to the first temperature after the addition of the seed material, takes

\*place at a rate of 0.2 - 3°C/min.

5. A method according to one of the preceding claims, characterized in that the method is carried out as a continuous process.

5 6. A method according to claim 5, characterized in that the mixture is divided into a first relatively small stream and a second relatively large stream, wherein the first stream is cooled more slowly than the second stream, and subsequently used as seed material, whereas  
10 the second stream is cooled yielding solid chocolate.



(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
1 February 2001 (01.02.2001)

PCT

(10) International Publication Number  
**WO 01/06863 A1**

- (51) International Patent Classification<sup>7</sup>: **A23G 1/18**
- (21) International Application Number: **PCT/NL00/00525**
- (22) International Filing Date: **24 July 2000 (24.07.2000)**
- (25) Filing Language: **Dutch**
- (26) Publication Language: **English**
- (30) Priority Data:  
1012691 ✓ 23 July 1999 (23.07.1999) **NL**
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- (81) Designated States (national): **AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.**
- (84) Designated States (regional): **ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).**
- Published:**  
— *With international search report.*
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: **METHOD FOR THE MANUFACTURE OF CHOCOLATE**

(57) Abstract: The invention relates to a method for the manufacture of chocolate, wherein liquid chocolate mass is mixed with seed material, and the mixture is cooled to below the melting temperature. According to the invention the seed material mixture is used having a temperature of at least 30 °C, and which has not exceeded the critical temperature. A chocolate manufactured in this manner will exhibit less or no fat bloom during prolonged storage.

**WO 01/06863 A1**

## Method for the manufacture of chocolate

The present invention relates to a method comprising

a) the preparation of a cooled but still liquid chocolate mass which comprises i) a fat selected from cocoa butter and cocoa butter equivalents (CBE), and at least one component selected from a) sugar, b) cocoa mass and c) cocoa powder,

b) mixing the liquid chocolate mass with a seed material, and

10 c) allowing the mixture to cool to a first temperature below the melting temperature of chocolate, producing solid chocolate,

the seed material used in step b) being cooled mixture.

15 Such a method is known from the European patent application 0 765 606. This describes how chocolate mass from a vessel is subjected to ultrasonic treatment in order to form stable  $\beta$  polymorph crystals. A portion of the thus-treated mass is cooled and returned to the vessel.

20 The disadvantage of this method is that an apparatus is required for the generation of ultrasound for the removal of unstable polymorph crystals formed with the method, while in addition to this outlay also raising the energy costs during production of a chocolate product.

25 The object of the present invention is to provide a method according to the preamble with which the disadvantages are to large extent eliminated. It is the particular object of the invention to provide a method producing such a chocolate, which includes a chocolate product, which during prolonged storage will develop no or only a slight amount of white efflorescence (will exhibit less or no fat bloom).

35 To this end the method according to the present invention is characterized in that when preparing the

liquid chocolate mass, it is heated to above the critical temperature, and subsequently cooled to a second temperature between the first temperature and the critical temperature, the thus cooled chocolate mass is mixed with  
5 the seed material, the seed material used being a cooled mixture at a temperature above 30°C, but which substance has not exceeded the critical temperature and which substantially does not contain any crystalline material in the  $\beta'$  phase, and in that to produce solid chocolate, the  
10 mixture is subsequently cooled to the first temperature.

Thus a simplified method is provided for the preparation of chocolate which during prolonged storage will exhibit less fat bloom or none at all. Compared with the known method, the energy consumption is limited. In  
15 the present application the critical temperature is the temperature at which all forms of crystalline fat have changed to the molten state. This temperature may be determined by melting a sample whose fat is in the  $\beta$  condition (obtained, for example, by leaving molten  
20 chocolate for at least three days at a temperature of 22°C) and to heat it to a temperature X and to maintain it for one minute at that temperature. The mass is subsequently cooled to 23°C at a rate of 1°C/min. (while avoiding that the liquid chocolate mass comes into contact  
25 with a surface whose temperature is more than 3°C lower than that of the chocolate mass), and examined to see whether the  $\beta$  or the  $\beta'$  phase develops. This experiment is carried out mechano-statically. That temperature X is the critical temperature with which after cooling again solid  
30 chocolate is obtained whose crystallization phase is substantially  $\beta'$ . The melting point of chocolate depends to some extent on the rate of cooling during production. Moreover, the melting point is not one single value, because chocolate has a melting range of several degrees.  
35 Indications of temperature with regard to melting temperature are in the present application related to the lowest value of the melting range. For a reliable process, the liquid chocolate mass will generally be heated to at

least 2°C, preferably to at least 5°C above the critical temperature. A higher temperature shortens the time during which the chocolate mass has to be heated above the critical temperature. In the present application, the first temperature is understood to be the temperature at which molten chocolate mass is solidified. This temperature is below the melting temperature of the chocolate. The second temperature, which may also be called the mixing temperature, is suitably at least 2°C below the critical temperature and at least 2°C above the first temperature, conveniently above the melting temperature of the chocolate. Cocoa butter is preferably mixed at a temperature in the vicinity of  $T_{opt}$  obtained with the (empirical) formula:

$$T_{opt} = 1.44 * [St] - 3.3 * [Ar] - 6$$

[St] being the concentration of stearic acid and [Ar] the concentration of arachidic acid as present in the free ester form in cocoa butter. When the present application refers to a substance not having exceeded the critical temperature, this means counting from the last time the substance is at least partially in a crystalline ( $\beta$ ) phase.

As an alternative to ultrasonic treatment it is known in the art to add seed crystals to a liquid chocolate mass (Hachiya, I., et al. in Seeding Effects on Solidification Behaviour of Cocoa Butter and Dark Chocolate. II. Physical Properties of Dark Chocolate, in *J. Amer. Oil Chem. Soc.* 66:1763-1770 (1989)).

Such a method has several disadvantages. First, solid chocolate has to be ground into (preferably the smallest possible) seed crystals. Second, said seed crystals should be mixed as homogeneously as possible with the liquid chocolate mass. Until now, these disadvantages have prevented the application of this method in large-scale production of chocolate, which term includes in the present invention also chocolate-comprising products such as biscuits with chocolate, and the like. Preferably, in the cooling steps, the temperature of the wall (that is to

say the interior wall) is suitably at most 5°C lower than the temperature of the optionally seeded or unseeded liquid chocolate mass, preferably at most 3°C lower, and most preferably at most 2°C.

- 5 This ensures that the fat in the solid chocolate will not be in the undesirable  $\beta'$  phase.

The preparation of chocolate has been widely researched. This has also involved fundamental research on the behaviour of its components such as the  
10 crystallization behaviour of cocoa butter. Schlichter-Aronhime, J. et al. (ref.1) described the formation of stable crystalline seed material in a melt. This may be done by alternating the temperature causing low-melting crystals to redissolve while more stable crystals are  
15 left. Hence, the method described in said (and other) publications relates to cocoa butter in non-stirred (static) conditions. The above publication does not concern a liquid chocolate mass which after all contains in addition a sweetener such as sugar and optionally cocoa  
20 powder. It is well known in the art that these factors affect the crystallization behaviour (of fat) in the chocolate. Ref. 2, for example, describes the differences between static and dynamic formation of chocolate. Refs. 3 and 4 describe the effects of other components on the  
25 formation of chocolate, and in particular that these may have a considerable effect on the same.

To start the process of a continuous production, cocoa butter may be used as the seed material, as prepared according to Ref. 5, whereas subsequently mixture cooled  
30 to approximately the first temperature but having a temperature of at least 30°C, is used as seed material.

Consequently, once production is started up, this seed material is available in liberal quantities since it can be obtained just prior to the mixture cooling to below  
35 the first temperature. In accordance with an alternative embodiment, mixture that is cooled to below the first temperature may be remelted, taking care not to exceed the critical temperature. In each of the cases the seed

material is added at a temperature lower than the critical temperature and at a temperature of at least 30°C, such as suitably at least 32°C. In practice, the seed material will be added at a temperature that is the same as, or  
5 lower than the second temperature. The temperature may advantageously be chosen such that it contributes to the further cooling of the liquid chocolate mass.

The quantity of liquid substance being added is preferably 10 - 20% by volume of the fat content of the  
10 final mixture.

Although the amount of liquid substance added as seed material may vary greatly, for example between 5 and 90%, the above-mentioned preferred range of percentages will provide a method combining a large production volume with  
15 limited sensitivity to variations in the production conditions, such as changes in the composition of components and temperature variations within the installation where the method is applied.

Prior to being mixed with the seed material, it is  
20 advantageous for the liquid chocolate mass to be cooled to a second temperature of at least 4°C below the critical temperature.

This provides a robust process that is less sensitive to temperature deviations.

25 Advantageously, cooling to the first temperature takes place subsequent to the addition of the seed material, at a rate of 0.2 - 3°C/min.

Preferably the method is carried out as a continuous process.

30 Continuous process simplifies the method to a considerable extent, especially since seed material can be added in a simple manner from downstream product streams.

According to a preferred embodiment the mixture is divided into a first relatively small stream and a second  
35 relatively large stream, wherein the first stream is cooled more slowly than the second stream and subsequently used as seed material, whereas the second stream is cooled yielding solid chocolate.

Cooling the small stream that is used as the seed material more slowly, ensures that high-quality seed material is obtained and allows the second stream to be cooled relatively quickly yielding solid chocolate with the desired qualities.

The present invention will now be elucidated with reference to the following exemplary embodiments.

#### Example

For all the experiments and the control experiments chocolate was prepared consisting of 9.0% defatted cocoa powder (natural, fat content < 0.5%), 35.0% cocoa butter (soft butter from Bahia, iodine value 41, having the fat composition shown in table I), 55.5% fine powder sugar and 0.5% lecithin.

TABLE I

Iodine value      triglycerides      fatty acids

Iodine value	Triglycerides		Fatty acids	
		%		%
40,7	C48	0,3	C16:0	23,5
	C50	16,4	C16:1	0,3
	C52	44,7	C18:0	31,8
	C54	36,6	C18:1	38,8
	C56	2,0	C18:2	4,1
			C18:3	0,3

20

The iodine value is determined by the Wijs method (IUPAC method 2.205). The triglyceride composition is determined by GLC (IUPAC method 2.323) and the fatty acid composition is determined with the aid of GLC via fatty acid methyl esters (IUPAC methods 2.301 and 2.302).

EXAMPLE I

For the preparation of 750 g chocolate the cocoa powder and the sugar were mixed, and heated to 60°C in an oven. To this mixture a portion of the warm cocoa butter is admixed, is yielding a chocolate mass with a fat content  
5 of 25%. This chocolate mass is rolled with 700-800 kPa/cm<sup>2</sup> at approximately 30°C in order to reduce the cocoa particles and sugar, and reheated to 60°C and mixed and again rolled, now with 900-1000 kPa/cm<sup>2</sup> at approximately 30°C. More cocoa butter is added such that the mixture  
10 contains 80% of the total quantity of cocoa butter. Immediately after rolling and the addition of more cocoa butter the chocolate mass is heated for 30 minutes at 60°C, yielding a liquid chocolate mass. The temperature of 60°C is 20-22°C above the critical temperature.

15 The remaining cocoa butter (the cocoa butter used had solidified through natural cooling, and had been stored for certainly more than three days) is molten together with the lecithin and under stirring heated to 34°C. This is done in a vessel having a wall temperature of 35°C in  
20 order to ensure that none of the cocoa butter reaches a temperature above the critical temperature.

The liquid chocolate mass is cooled to 34°C (wall temperature above 26°C) and the mixture of cocoa butter and lecithin is admixed with the liquid chocolate mass.  
25 The mixture thus obtained is cooled under stirring to 26°C in a vessel with a wall temperature of 26°C. This temperature is below the melting temperature of the chocolate (melting range 30.1-34.5°C. The thus cooled chocolate is immediately poured into moulds that have been  
30 heated to 26°C, vibrated to remove the air bubbles, and subsequently kept at 26°C for 1.5 hours. The filled moulds are then stored for 30 minutes at 10°C to facilitate the removal of the chocolate from the moulds. After removal, the chocolate is wrapped in aluminium foil.

35

#### EXAMPLE II

A quantity of material to be rolled (composition: minimum total content of cocoa 25% (of which at least 6.7%



defatted dry cocoa and at least 18% cocoa butter), at least 21.5% of milk constituents (of which at least 7.5% milk at) and at least 46% sucrose)) is heated to 55-60°C in an oven (mixture A). Subsequently it is cooled to 36°C  
5 on a water bath of 36°C.

Separately, 65.8 g of cocoa butter and 2.5% lecithin are mixed and heated to 36°C (mixture B).

Mixture A and 54 g of mixture B are combined at 36°C and cooled to 25°C. After 30 minutes material poured into  
10 moulds is cooled to 10°C.

The addition of partially cooled mixture B (or product molten again to at least 30°C) to a mixture A, results in chocolate products that exhibit the same favourable fat bloom characteristics as chocolate obtained  
15 directly from mixture A and mixture B.

References

- 1) Schlichter-Aronhime, J. et al. Solidification and Polymorphism in Cocoa Butter and the Blooming Problems, in *Crystallization and Polymorphism of Fats and Fatty Acids*, Surfactant Science Series, Vol. 31, edited by N. Garti and K. Sato, Marcel Dekker Inc., New York, pp. 363-393, (1988)
- 2) Loisel, C. et al. Dynamic Crystallization of Dark Chocolate as Affected by Temperature and Lipid Additives in *Journal of Food Science* 63 (1), pp 73-79, (1998)
- 3) Seguire, E. S., Tempering, the inside story, *Manufact. Conf.* 71:117-125 (1991)
- 4) Bricknell J. et al. Relation of Fat Bloom in Chocolate to Polymorphic Transition in Cocoa Butter, *JAACS*, Vol. 75, No. 1, pp 1609-1615, (1998)
- 5) Adenier, H. et al., Solidification and Polymorphism in Cocoa Butter and the Blooming problems, *Ind. Aliment.* Vol. 4, p 315 (1978)

CLAIMS

1. A method for the manufacture of chocolate, which method comprises

a) the preparation of a cooled but still liquid chocolate mass which comprises i) a fat selected from  
5 cocoa butter and cocoa butter equivalents (CBE), and at least one component selected from a) sugar, b) cocoa mass and c) cocoa powder,

b) mixing the liquid chocolate mass with a seed material, and

10 c) allowing the mixture to cool to a first temperature below the melting temperature of chocolate, producing solid chocolate,

the seed material used in step b) being cooled mixture, characterized in that when preparing the liquid  
15 chocolate mass, it is heated to above the critical temperature, and subsequently cooled to a second temperature between the first temperature and the critical temperature, the thus cooled chocolate mass is mixed with the seed material, the seed material used being a cooled  
20 mixture at a temperature above 30°C, but which substance has not exceeded the critical temperature and which substantially does not contain any crystalline material in the  $\beta'$  phase, and in that to produce solid chocolate, the mixture is subsequently cooled to the first temperature.

25 2. A method according to claim 1, characterized in that the quantity of liquid substance being added is 10 - 20% by volume of the fat content of the final mixture.

3. A method according to claim 1 or 2,  
30 characterized in that prior to being mixed with the seed material, the liquid chocolate mass is cooled to a second temperature of at least 4°C below the critical temperature.

4. A method according to one of the preceding  
35 claims, characterized in that cooling to the first temperature after the addition of the seed material, takes

place at a rate of 0.2 - 3°C/min.

5. A method according to one of the preceding claims, characterized in that the method is carried out as a continuous process.

5 6. A method according to claim 5, characterized in that the mixture is divided into a first relatively small stream and a second relatively large stream, wherein the first stream is cooled more slowly than the second stream, and subsequently used as seed material, whereas  
10 the second stream is cooled yielding solid chocolate.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/NL 00/00525

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23G1/18

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, FSTA

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 26 02 877 A (WINKLER DUENNEBIER KG MASCH;DOMGRAFF AUTOMATION) 28 July 1977 (1977-07-28) page 3, line 1 -page 10, line 14; claims ---	1-6
X	EP 0 765 606 A (KRAFT JACOBS SUCHARD R & D INC) 2 April 1997 (1997-04-02) page 7, line 15 - line 21 claims 13-24; examples ---	1-6
X	WO 98 30108 A (MARS INC) 16 July 1998 (1998-07-16) page 32, line 11 -page 60, line 17; examples ---	1-6
X	US 4 283 436 A (SOETERS CORNELIS J ET AL) 11 August 1981 (1981-08-11) column 13, line 41 -column 15, line 25 ---	1,3-6
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search

7 November 2000

Date of mailing of the international search report

14/11/2000

Name and mailing address of the ISA

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NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Boddaert, P

## INTERNATIONAL SEARCH REPORT

Intern. Application No  
PCT/IL 00/00525

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 521 205 A (UNILEVER PLC) 7 January 1993 (1993-01-07) the whole document ----	1-6
A	EP 0 768 038 A (FUJI OIL CO LTD) 16 April 1997 (1997-04-16) page 4, line 41 -page 5, line 14; examples ----	1
A	KOYANO T ET AL: "FAT POLYMORPHISM AND CRYSTAL SEEDING EFFECTS ON FAT BLOOM STABILITY OF DARK CHOCOLATE" FOOD STRUCTURE,US,SCANNING MICROSCOPY INTERNATIONAL, CHICAGO, IL, vol. 9, 1 January 1990 (1990-01-01), pages 231-240, XP002061521 ISSN: 1046-705X the whole document ----	1-6
A	GB 2 048 928 A (RAU LEBENSMITTELWERKE) 17 December 1980 (1980-12-17) ----	
A	EP 0 496 310 A (BATTELLE MEMORIAL INSTITUTE) 29 July 1992 (1992-07-29) -----	

# INTERNATIONAL SEARCH REPORT

International patent family members

International Application No

PCT/IL 00/00525

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
DE 2602877	A	28-07-1977	NONE	
EP 0765606	A	02-04-1997	NONE	
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**PCT****REQUEST**

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

**RECORD COPY**Confirmation  
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**PCT/NL**

International Application No.

**00/00525****24 JUL 2000**

International Filing Date

**(24.07.00)****BUREAU VOOR DE INDUSTRIËLE EIGENDOM  
P.C.T. INTERNATIONAL APPLICATION**

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference

(if desired) (12 characters maximum) **WO 800148-AI****Box No. I TITLE OF INVENTION**

Method for the manufacture of chocolate

**Box No. II APPLICANT**

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

Stichting voor de Technische Wetenschappen  
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☐ This person is also inventor.

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**+31 30 600 13 19**

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State (that is, country) of nationality:

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**NL**This person is applicant  
for the purposes of:☐ all designated  
States☒ all designated States except  
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of America only☐ the States indicated in  
the Supplemental Box**Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)**

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

Van Malssen, Kees Frederik  
Lepelblad 111  
NL-1441 VH PURMEREND  
the Netherlands

This person is:

☐ applicant only☒ applicant and inventor☐ inventor only (If this check-box  
is marked, do not fill in below.)

State (that is, country) of nationality:

**NL**

State (that is, country) of residence:

**NL**This person is applicant  
for the purposes of:☐ all designated  
States☐ all designated States except  
the United States of America☒ the United States  
of America only☐ the States indicated in  
the Supplemental Box☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.**Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**The person identified below is hereby/has been appointed to act on behalf  
of the applicant(s) before the competent International Authorities as:☒ agent☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

ALTENBURG, Bernardus Stephanus Franciscus et al.  
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☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.



## Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

*If none of the following sub-boxes is used, this sheet should not be included in the request.*

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

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This person is:

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☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:  
NL

State (that is, country) of residence:  
NL

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

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Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

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Parklaan 1a  
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the Netherlands

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☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:  
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State (that is, country) of residence:  
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This person is applicant for the purposes of:

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Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

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the Netherlands

This person is:

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☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:  
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## Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

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- ☒ **AP ARIPO Patent:** GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
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- ☒ **OA OAPI Patent:** BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT. *Other kind of protection or treatment desired, specify on dotted line*

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| <input checked="" type="checkbox"/> KG Kyrgyzstan                            | <input checked="" type="checkbox"/> ZW Zimbabwe                                  |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea |  |
| <input checked="" type="checkbox"/> KR Republic of Korea                     |  |
| <input checked="" type="checkbox"/> KZ Kazakhstan                            |  |
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Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

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**Precautionary Designation Statement** In addition to the designations made above the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn. *(Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)*

<b>Box No. VI PRIORITY CLAIM</b>		<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.		
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application:* regional Office	international application: receiving Office
item (1) 23 July, 1999 (23.07.99)	1012691	NL		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office* identified above as item(s).<sup>1</sup>

\* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

### Box No. VII INTERNATIONAL SEARCHING AUTHORITY

**Choice of International Searching Authority (ISA)** (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):

ISA /

**Request to use results of earlier search; reference to that (if earlier search has been carried out by or requested from the International Searching Authority):**

Date (day/month/year)

Number

Country (or regional Office)

### Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following number of sheets:

request : 4

description (excluding sequence listing part) : 23

claims : 2

abstract : 1

drawings :

sequence listing part of description :

Total number of sheets 14

This international application is accompanied by the item(s) marked below:

1. ☒ fee calculation sheet

2. ☐ separate signed power of attorney

3. ☐ copy of general power of attorney; reference number, if any:

4. ☐ statement explaining lack of signature

5. ☐ priority document(s) identified in Box No. VI as item(s):

6. ☐ translation of international application into (language):

7. ☐ separate indications concerning deposited microorganism or other biological material

8. ☐ nucleotide and/or amino acid sequence listing in computer readable form

9. ☒ other (specify): Copy of Search Report

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: NL

### Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

ALTENBURG, Bernardus Stephanus Franciscus et al.

Amsterdam, 21 July, 2000

For receiving Office use only		2. Drawings:  <input type="checkbox"/> received:  <input type="checkbox"/> not received:
1. Date of actual receipt of the purported international application:	24 JUL 2000 (24.07.00)	
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): ISA /	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.	

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Date of receipt of the record copy by the International Bureau:	23 AUGUST 2000 (23.08.00)

WO 800148-A1/ho

Werkwijze voor het bereiden van chocolade.

De onderhavige uitvinding heeft betrekking op een werkwijze welke omvat

a) het bereiden van een afgekoelde maar nog vloeibare chocolademassa, welke i) een vet gekozen uit cacao-  
5 boter en cacaoboterequivalenten (CBE), en ii) ten minste een component gekozen uit a) suiker, b) cacaomassa en c) cacao-poeder, omvat,

b) het mengen van de vloeibare chocolademassa met entmateriaal, en

10 c) het laten afkoelen van het mengsel tot een eerste temperatuur gelegen onder de smelttemperatuur van de chocolade, onder oplevering van vaste chocolade,

waarbij als entmateriaal in stap b) afgekoeld mengsel wordt gebruikt.

15 Een dergelijke werkwijze is bekend uit de Europese octrooiaanvraag 0 765 606. Hierin wordt beschreven hoe uit een houder afkomstige chocolademassa wordt onderworpen aan een behandeling met ultrasoon geluid teneinde stabiele  $\beta$  polymorfe kristallen te vormen. Een deel van aldus behandelde  
20 massa wordt afgekoeld en teruggevoerd naar de houder.

Deze werkwijze heeft als nadeel dat een inrichting vereist is voor het opwekken van ultrasoon geluid teneinde bij de werkwijze ontstane instabiele polymorfe kristallen te verwijderen, terwijl naast deze investering ook de energie-  
25 kosten tijdens de productie van een chocoladeproduct verhoogd zijn.

De onderhavige uitvinding beoogt een werkwijze van de in de aanhef genoemde soort te verschaffen waarmee de nadelen in vergaande mate worden opgeheven. De uitvinding  
30 beoogt in het bijzonder een dergelijke werkwijze te verschaffen waarmee een chocolade, daaronder begrepen chocoladeproduct, wordt verschaft welke bij langdurige opslag nauwelijks of niet wit uitslaat (derhalve minder of geen vetrijp vertoont).

35 Hiertoe wordt de werkwijze volgens de onderhavige uitvinding gekenmerkt doordat de vloeibare chocolademassa bij

het bereiden ervan tot boven de kritische temperatuur wordt verwarmd, en vervolgens wordt afgekoeld tot een tweede temperatuur gelegen tussen de eerste temperatuur en de kritische temperatuur, de aldus afgekoelde chocolademassa met entmateriaal wordt gemengd waarbij als entmateriaal afgekoeld mengsel wordt gebruikt met een temperatuur boven de 30°C maar welke substantie de kritische temperatuur niet heeft overschreden en in hoofdzaak geen kristallijn materiaal in de  $\beta'$  toestand bevat, en het mengsel voor het maken van vaste chocolade vervolgens wordt afgekoeld tot de eerste temperatuur.

Aldus wordt een vereenvoudigde werkwijze verschaft waarmee een chocolade wordt bereid die bij langdurige opslag geen of verminderde vetrijp vertoont. Hierbij zijn de energiekosten in vergelijking met de bekende werkwijze beperkt. In de onderhavige aanvraag is de kritische temperatuur die temperatuur waarbij alle vormen van kristallijn vet in de gesmolten toestand zijn overgegaan. Deze temperatuur kan worden bepaald door een monster waarvan het vet zich in de  $\beta$  toestand bevindt (bijvoorbeeld gevormd door gesmolten chocolade gedurende ten minste 3 dagen bij 22°C te laten staan) te smelten en te verwarmen tot een temperatuur X en gedurende 1 min. bij die temperatuur te houden. Daarna wordt met een snelheid van 1°C/min. tot 23°C afgekoeld (waarbij wordt vermeden dat de vloeibare chocolademassa in contact staat met een oppervlak dat een meer dan 3°C lagere temperatuur heeft dan de chocolademassa) en onderzocht of de  $\beta$  of de  $\beta'$  toestand wordt gevormd. Dit experiment wordt mechanisch-statisch uitgevoerd. Die temperatuur X is de kritische temperatuur, waarmee na weer afkoelen vaste chocolade wordt verkregen waarvan de kristallisatietoestand in hoofdzaak  $\beta'$  is. Het smeltpunt van chocolade hangt enigszins af van de snelheid waarmee deze bij de vorming ervan is afgekoeld. Ook is het smeltpunt geen enkele waarde; chocolade vertoont een smelttraject van enkele graden. Wanneer in de onderhavige aanvraag wordt gesproken over temperatuuraanduidingen ten opzichte van de smelttemperatuur, dan zijn deze gerelateerd aan de benedenwaarde van het smelttraject. In het algemeen zal voor een betrouwbare procesvoering de vloeibare chocolademassa ten

minste 2°C, bij voorkeur ten minste 5°C boven de kritische temperatuur worden verwarmd. Een hogere temperatuur bekort de tijd gedurende welke de chocolademassa boven de kritische temperatuur moet worden verwarmd. In de onderhavige aanvraag  
5 wordt onder de eerste temperatuur de temperatuur verstaan waarbij gesmolten chocolademassa wordt gestold. Deze temperatuur ligt dus beneden de smelttemperatuur van de chocolade. De tweede temperatuur, welke ook kan worden aangeduid als de mengtemperatuur, ligt geschikt ten minste 2°C beneden de  
10 kritische temperatuur en ten minste 2°C boven de eerste temperatuur, gerieflijk boven de smelttemperatuur van de chocolade. Voor cacaoboter geschiedt het mengen bij voorkeur nabij een temperatuur  $T_{opt}$  die wordt gegeven door de (empirische) formule:

15 
$$T_{opt} = 1,44 \cdot [St] - 3,3 \cdot [Ar] - 6$$

waarbij [St] de concentratie stearinezuur en [Ar] de concentratie arachidezuur zijn, zoals deze in vrije en estervorm in cacaoboter aanwezig zijn. Wanneer in de onderhavige aanvraag wordt gesproken over een substantie die de kritische temperatuur niet heeft overschreden, dan dient dit te worden gerekend vanaf de laatste keer dat de substantie zich ten minste gedeeltelijk in een kristallijne ( $\beta$ ) toestand bevindt.

Als alternatief voor een behandeling met ultrasoon geluid is het in het vak (Hachiya, I., et al. in Seeding  
25 Effects on Solidification Behavior of Cocoa Butter and Dark Chocolate. II. Physical Properties of Dark Chocolate, in *J. Amer. Oil Chem. Soc.* 66:1763-1770(1989) bekend om entkristallen aan een vloeibare chocolademassa toe te voegen.

Een dergelijke werkwijze heeft verscheidene nadelen.  
30 Ten eerste dient vaste chocolade tot (liefst zo klein mogelijke) entkristallen te worden vermalen. Ten tweede moeten deze entkristallen zo homogeen mogelijk door de vloeibare chocolademassa worden gemengd. Deze nadelen hebben tot nu toe toepassing van deze werkwijze bij de grootschalige productie  
35 van chocolade, waaronder in de onderhavige uitvinding tevens chocolade omvattende producten zoals koekjes met chocolade en dergelijke worden begrepen, verhinderd.

Bij voorkeur is bij de afkoelstappen de temperatuur van de wand (dat wil zeggen de binnenwand) geschikt ten

hoogste 5°C lager dan de temperatuur van de al dan niet reeds beënte vloeibare chocolademassa, bij voorkeur ten hoogste 3°C lager en met meer voorkeur ten hoogste 2°C.

5 Aldus wordt met meer zekerheid bevorderd dat het vet in de gevormde vaste chocolade zich niet in de ongewenste  $\beta'$  toestand bevindt.

Er is naar de bereiding van chocolade veel onderzoek verricht. Ook is hierbij fundamenteel onderzoek verricht naar het gedrag van bestanddelen daarvan, zoals het kristallisa-  
10 tiegedrag van cacaoboter. Schlichter-Aronhime, J. et al. (ref. 1) beschrijven de vorming van stabiel kristallijn entmateriaal in een smelt. Dit kan geschieden door het wisselen van de temperatuur waarbij laag-smeltende kristallen weer oplossen en stabielere kristallen overblijven. De in deze  
15 publicatie, en ook elders, beschreven werkwijze heeft derhalve betrekking op cacaoboter onder niet-geroerde (statische) omstandigheden. Het betreft hier derhalve geen vloeibare chocolademassa welke immers verder een zoetstof zoals suiker en optioneel cacaopoeder omvat. Het is in het vak welbekend  
20 dat deze factoren van invloed zijn op het kristallisatiegedrag (van vet) in de chocolade. Zo beschrijft ref. 2 het optreden van verschillen tussen statische en dynamische vorming van chocolade. Ref. 3 en 4 beschrijven het effect van andere componenten op de vorming van chocolade, en in het  
25 bijzonder dat deze een zeer grote invloed daarop kunnen hebben.

Voor het opgang brengen van een continue productie kan als het entmateriaal cacaoboter worden gebruikt zoals bereid volgens ref. 5, terwijl daarna als entmateriaal tot  
30 nabij de eerste temperatuur afgekoeld maar met een temperatuur van ten minste 30°C mengsel wordt gebruikt.

Dit entmateriaal is dus, zodra de productie éénmaal is opgestart, in ruime mate voorhanden, aangezien het kan worden verkregen juist voordat het mengsel tot beneden de  
35 eerste temperatuur wordt afgekoeld. Volgens een alternatieve uitvoeringsvorm kan tot beneden de eerste temperatuur afgekoeld mengsel weer worden opgesmolten, waarbij er voor wordt gemaakt dat de kritische temperatuur niet wordt overschreden. In elk van de gevallen wordt het entmateriaal toegevoegd met

een temperatuur beneden de kritische temperatuur en ten minste 30°C, zoals geschikt ten minste 32°C. In de praktijk zal het entmateriaal worden toegevoegd bij een temperatuur gelijk aan of lager dan de tweede temperatuur. Hierbij kan met voordeel de temperatuur van het entmateriaal zodanig zijn gekozen dat dit bijdraagt aan het verder afkoelen van de vloeibare chocolademassa.

Bij voorkeur omvat de hoeveelheid vloeibare substantie die wordt toegevoegd 10 - 20 vol.% van het vet van het uiteindelijke mengsel.

Ofschoon de hoeveelheid vloeibare substantie die als entmateriaal wordt toegevoegd binnen een groot bereik kan worden gevarieerd, bijvoorbeeld tussen 5 en 90%, wordt binnen het genoemde voorkeursbereik een werkwijze verschaft welke een groot productievolume combineert met beperkte gevoeligheid voor variaties in de procesomstandigheden, zoals wisselende grondstofsamenstellingen en temperatuurvariaties binnen de inrichting waarin de werkwijze wordt toegepast.

Met voordeel wordt de vloeibare chocolademassa tot een tweede temperatuur afgekoeld die ten minste 4°C beneden de kritische temperatuur ligt alvorens met entmateriaal te worden gemengd.

Aldus wordt een robuust proces verschaft dat minder gevoelig is voor temperatuurafwijkingen.

Met voordeel geschiedt het afkoelen na het toevoegen van entmateriaal tot de eerste temperatuur met een snelheid van 0,2 tot 3°C/min.

Bij voorkeur wordt de werkwijze als een continu-proces bedreven.

Continu bedrijf vereenvoudigt de werkwijze aanmerkelijk, in het bijzonder doordat op eenvoudige wijze entmateriaal kan worden toegevoegd afkomstig van benedenstroomse productstromen.

Volgens een voorkeursuitvoeringsvorm wordt het mengsel gesplitst in een eerste relatief kleine stroom en een tweede relatief grote stroom, waarbij de eerste stroom langzamer wordt afgekoeld dan de tweede stroom en vervolgens als entmateriaal wordt gebruikt en de tweede stroom wordt afgekoeld onder oplevering van vaste chocolade.



Door de kleine stroom, die als entmateriaal wordt gebruikt, langzamer af te koelen, wordt verzekerd dat kwalitatief goed entmateriaal wordt verkregen en kan de tweede stroom relatief snel worden afgekoeld onder oplevering van  
5 vaste chocolade met de gewenste eigenschappen.

De onderhavige uitvinding zal thans worden toegelicht aan de hand van de volgende uitvoeringsvoorbeelden.

#### Voorbeeld

Voor de experimenten en controle-experimenten werd chocolade  
10 bereid bestaande uit 9,0% ontvet cacaopoeder (type naturel, vetpercentage <0,5%), 35,0% cacaoboter (zachte boter uit Bahia, joodgetal 41, met de in tabel I weergegeven vetsamenstelling), 55,5% fijne poedersuiker en 0,5% lecithine.

TABEL I

Joodgetal triglyceriden vetzuren

5	Joodgetal	Triglyceriden	vetzuren
		%	%
	40,7	C48 0,3	C16:0 23,5
		C50 16,4	C16:1 0,3
		C52 44,7	C18:0 31,8
		C54 36,6	C18:1 38,8
10		C56 2,0	C18:2 4,1
			C18:3 0,3

Het joodgetal is bepaald met de Wijs-methode (IUPAC methode 2.205). De triglyceridesamenstelling is bepaald met GLC (IUPAC methode 2.323) en de vetzuursamenstelling met behulp van GLC via vetzuurmethylesters (IUPAC methoden 2.301 en 2.302).

## VOORBEELD I

Voor de bereiding van 750 g chocolade, worden het cacaopoeder en de suiker gemengd en in een oven tot 60°C verwarmd. Aan dit mengsel wordt een deel van de cacaoboter warm toegevoegd en gemengd, onder oplevering van een chocolademassa die 25% vet bevat. Voor het verkleinen van cacao-deeltjes en suiker wordt deze chocolademassa gewalst met 700-800 kPa/cm<sup>2</sup> bij ca. 30°C weer tot 60°C verwarmd en gemengd en weer gewalst, nu met 900-1000 kPa/cm<sup>2</sup> en bij ca. 30°C. Meer cacaoboter wordt toegevoegd, zodanig dat het mengsel 80% van de totale hoeveelheid cacaoboter bevat. Direct na het walsen en toevoegen van de verdere cacaoboter wordt de chocolademassa gedurende een half uur bij 60°C verwarmd onder oplevering van een vloeibare chocolademassa. De temperatuur van 60°C ligt 20-22°C boven de kritische temperatuur.

De rest van de cacaoboter (als cacaoboter werd door natuurlijke afkoeling gestolde cacaoboter gebruikt die een onbepaalde periode van zeker meer dan 3 dagen heeft gestaan) wordt tezamen met de lecithine gesmolten en onder roeren tot 34°C verwarmd. Dit geschiedt in een houder met een wandtemperatuur van 35°C waardoor wordt verzekerd dat geen (deel van)

de cacaoboter een temperatuur bereikt die boven de kritische temperatuur ligt.

De vloeibare chocolademassa wordt afgekoeld tot 34°C (wandtemperatuur boven 26°C) en het mengsel van cacaoboter en lecithine wordt toegevoegd en met de vloeibare chocolademassa gemengd. Het aldus gevormde mengsel wordt onder roeren tot 26°C afgekoeld in een houder met een wandtemperatuur van 26°C. Deze temperatuur ligt beneden de smelttemperatuur (smelttraject 30,1-34,5°C) van de chocolade. De aldus afgekoelde chocolade wordt direct in op 26°C verwarmde vormen gegoten, ter verwijdering van luchtbellens getrild, en vervolgens gedurende 1,5 uur bij 26°C gehouden. Vervolgens worden de gevulde vormen een half uur bij 10°C opgeslagen teneinde het lossen van de chocolade uit de vormen te vergemakkelijken. Na het lossen wordt de chocolade verpakt in aluminiumfolie.

#### VOORBEELD II

Een hoeveelheid walsgoed (Samenstelling: totaal cacaogehalte minimaal 25% (waarvan ten minste 6,7% ontvette droge cacao en ten minste 18% cacaoboter), melkbestanddelen ten minimaal 21.5% (waarvan ten minste 7,5% melkvet) en sucrose minimaal 46%)) wordt in een oven tot 55-60°C verwarmd (mengsel A). Daarna wordt het op een waterbad van 36°C tot 36°C afgekoeld.

Apart daarvan worden 65,8 g cacaoboter en 2,5% lecithine gemengd en op 36°C gebracht (mengsel B).

Mengsel A en 54 g van mengsel B worden samengevoegd bij 36°C en afgekoeld tot 25°C. Na 30 min. wordt in vormen gegoten materiaal tot 10°C afgekoeld.

Wanneer deels afgekoeld mengsel B (of weer tot ten minste 30°C opgesmolten product) wordt toegevoegd aan een mengsel A worden daar chocoladeproducten mee verkregen die dezelfde gunstige verrijp-eigenschappen laten zien als direct uit mengsel A en mengsel B verkregen chocolade.

## Referenties

- 1) Schlichter-Aronhime, J. et al. Solidification and Polymorphism in Cocoa Butter and the Blooming Problems, in *Crystallization and Polymorphism of Fats and Fatty Acids*, Surfactant Science Series, Vol. 31, edited by N. Garti and K. Sato, Marcel Dekker Inc., New York, pp. 363-393, (1988)
- 2) Loisel, C. et al. Dynamic Crystallization of Dark Chocolate as Affected by Temperature and Lipid Additives in *Journal of Food Science* 63 (1), pp 73-79, (1998)
- 3) Seguine, E. S., Tempering, the inside story, *Manufact. Conf.* 71:117-125 (1991)
- 4) Bricknell J. et al. Relation of Fat Bloom in Chocolate to Polymorphic Transition in Cocoa Butter, *JAACS*, Vol. 75, no. 1, pp 1609-1615, (1998)
- 5) Adenier, H. et al., Solidification and Polymorphism in Cocoa Butter and the Blooming problems, *Ind. Aliment.* vol. 4 pp 315 (1978)

CONCLUSIES

1. Werkwijze voor het bereiden van chocolade, welke werkwijze omvat

a) het bereiden van een afgekoelde maar nog vloeibare chocolademassa, welke i) een vet gekozen uit cacao-  
5 boter en cacaoboterequivalenten (CBE), en ii) ten minste een component gekozen uit a) suiker, b) cacaomassa en c) cacao-poeder, omvat,

b) het mengen van de vloeibare chocolademassa met entmateriaal, en

10 c) het laten afkoelen van het mengsel tot een eerste temperatuur gelegen onder de smelttemperatuur van de chocolade, onder oplevering van vaste chocolade,

waarbij als entmateriaal in stap b) afgekoeld mengsel wordt gebruikt, met het kenmerk, dat de vloeibare chocolademassa bij het bereiden ervan tot boven de kritische  
15 temperatuur wordt verwarmd, en vervolgens wordt afgekoeld tot een tweede temperatuur gelegen tussen de eerste temperatuur en de kritische temperatuur, de aldus afgekoelde chocolademassa met entmateriaal wordt gemengd waarbij als entmateriaal  
20 afgekoeld mengsel wordt gebruikt met een temperatuur boven de 30°C maar welke substantie de kritische temperatuur niet heeft overschreden en in hoofdzaak geen kristallijn materiaal in de  $\beta'$  toestand bevat, en het mengsel voor het maken van vaste chocolade vervolgens wordt afgekoeld tot de eerste  
25 temperatuur.

2. Werkwijze volgens conclusie 1, met het kenmerk, dat de hoeveelheid vloeibare substantie die wordt toegevoegd  
10 - 20 vol.% van het vet van het uiteindelijke mengsel omvat.

30 3. Werkwijze volgens conclusie 1 of 2, met het kenmerk, dat de vloeibare chocolademassa tot een tweede temperatuur wordt afgekoeld die ten minste 4°C beneden de kritische temperatuur ligt alvorens met entmateriaal te worden gemengd.

35 4. Werkwijze volgens een der voorgaande conclusies, met het kenmerk, dat het afkoelen na het toevoegen van entma-

teriaal tot de eerste temperatuur geschiedt met een snelheid van 0,2 tot 3°C/min.

5. Werkwijze volgens een der voorgaande conclusies, met het kenmerk, dat de werkwijze als een continu-proces

5 wordt bedreven.

6. Werkwijze volgens conclusie 5, met het kenmerk, dat het mengsel wordt gesplitst in een eerste kleine stroom en een tweede relatief grote stroom, waarbij de eerste stroom langzamer wordt afgekoeld dan de tweede stroom en vervolgens  
10 als entmateriaal wordt gebruikt en de tweede stroom wordt afgekoeld onder oplevering van vaste chocolade.

UITTREKSEL

De uitvinding heeft betrekking op een werkwijze voor het bereiden van chocolade waarbij een vloeibare chocolademassa wordt gemengd met entmateriaal, en het mengsel tot beneden de smelttemperatuur van het mengsel wordt afgekoeld. Volgens de  
5 uitvinding wordt als entmateriaal dat een temperatuur heeft van ten minste 30°C en de kritische temperatuur niet heeft overschreden. Een met deze werkwijze bereide chocolade vertoont bij langdurige opslag geen of minder vetrijp.

# PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>WO 800148-AI</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/NL 00/ 00525</b>	International filing date (day/month/year) <b>24/07/2000</b>	(Earliest) Priority Date (day/month/year) <b>23/07/1999</b>
Applicant  <b>STICHTING VOOR DE TECHNISCHE WETENSCHAPPEN</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

### 1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.



## INTERNATIONAL SEARCH REPORT

International Application No

P L 00/00525

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23G1/18

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, FSTA

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 26 02 877 A (WINKLER DUENNEBIER KG MASCH;DOMGRAFF AUTOMATION) 28 July 1977 (1977-07-28) page 3, line 1 -page 10, line 14; claims ---	1-6
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Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

## \* Special categories of cited documents :

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- "E" earlier document but published on or after the international filing date
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"&" document member of the same patent family

Date of the actual completion of the international search

7 November 2000

Date of mailing of the international search report

14/11/2000

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## INTERNATIONAL SEARCH REPORT

International Application No

P/NL 00/00525

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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